

Cu-Si₃N₄ AMB-substrates for circuit boards in power electronics

A. Rost¹; M. Kuczynski²; E. Zschippang¹; P. Dold³; J. Hörig⁴; S. Mosch¹; S. dos Santos⁵; P. Gierth¹; J. Winhauer⁶; J. Pfeiffer⁷; J. Schilm¹; M. Herrmann¹

1 Fraunhofer IKTS, Dresden, Germany

2 AlzChem Trostberg GmbH, Trostberg, Germany

3 Fraunhofer-Einrichtung für Wertstoffkreisläufe und Ressourcenstrategie IWKS, Alzenau, Germany

4 Fraunhofer CSP, Halle (Saale), Germany

5 Aurubis Stolberg GmbH & Co. KG, Stolberg, Germany

6 PVA IVS, Wetttenberg, Germany

7 PVA LWT, Wetttenberg, Germany

Abstract

In power electronics, SiC semiconductors are used for high performance modules with high demands on packing densities, voltage or current and applications of high switching frequencies. Also, increased operating temperatures of 200 °C or even more are possible with this kind of wide bandgap semiconductors. These demands are also imposed on the circuit board for the SiC semiconductors. Conventional substrates, made of Al₂O₃ with copper joined in a DCB (direct copper bonding) process reaches the limits of strength, reliability and thermal conductivity. For high thermal conductivity, AlN might become an alternative. In terms of high strength, needed for the ability to withstand challenging thermocycles for power electronics, Si₃N₄ is the best suited material. Building up circuit boards with this material requires a shift in the joining process from DCB to AMB (active metal bonding). All steps of the value chain from powder to the final AMB-substrate are implemented in a project related, European development. Ranging from the powder producer AlzChem, the copper producer Aurubis, up to PVA TePla for joining technology and quality analysis.

1 Motivation

With the introduction of SiC semiconductors, the demands for circuit boards are increasing in terms of thermal conductivity, maximum operation temperature, as also the ability to withstand larger thermal cycles. Substrates made of Al₂O₃ no longer meet the requirements, especially thermal conductivity and strength. While AlN is an option for good thermal management, only Si₃N₄ offers additionally the required strength, ref. **Table 1**.

Table 1: Comparison of Al₂O₃ and Si₃N₄ [1]

	Thermal conductivity / W/m·K (@20 °C)	Bending strength / MPa
Al ₂ O ₃	25	200 – 300
AlN	180	300 – 350
Si ₃ N ₄	100	> 800

To realize Cu-Si₃N₄ substrates, the technology DCB is not suitable [2; 3] but AMB technology has to be applied, which especially due to silver-copper based active metal fillers forms more ductile joining regions between Copper and Si₃N₄ [4].

Commercial Cu-Si₃N₄ AMB-substrates can be obtained from Asian suppliers. Within the German Project CuSiN we demonstrate a European based approach for the whole value chain. Starting with modified raw materials, Si₃N₄ powders having low impurity content and improved sinter ability have been developed. Powder processing and sinter regime were adapted to achieve large, homogenous and dense ceramic blocks for substrates with high thermal

conductivity and strength. The process of multi wire cutting, known from silicon wafering, was adapted to the ceramic material Si₃N₄ which is by orders of magnitude harder to process to substrates with a thickness of 300 µm. New copper qualities were investigated in Cu-Si₃N₄-AMB-substrates in combination with silver-based and also silver-free brazing processes to get high joining forces between metal and ceramic. For manufacturing AMB-substrates in the dimensions of mastercards in an industry scale, a concept for a graphite based vacuum brazing furnace was developed. Also based on graphite, a modular, space-saving batch carrier with a capacity for 500 AMB-substrates in mastercard format was developed and tested.

2 Results

2.1 Si₃N₄ Powder for high quality substrates

The task for AlzChem was to develop an improved or application-specific silicon nitride powder raw material for ceramics used in high-performance power electronics and e-mobility applications. The target parameter is an increase in thermal conductivity in sintered and finished ceramic with consistently high strength and electronic properties.

The main parameters influencing the achievement of high thermal conductivity are the following two. First the aluminum content in the starting powder, whereby contents of less than 0.01 % should be realized and gradations in the aluminum content. This parameter can be adjusted via the raw material and through suitable processing. The

modified raw material requires the development of adapted nitriding regimes and preparation processes. The second parameter is the phase fraction of β - Si_3N_4 and the crystallite size. These two factors control the sintering behavior and grain growth. In particular, a proportion of approx. 5 % coarse β - Si_3N_4 crystallites in the powder can lead to increased grain growth. As a result, the necessary grain size for high thermal conductivity can be achieved with short sintering times. Several powder mixtures of α -rich and β -rich powders were prepared for this purpose.

The material Silzot[®] SQ is a high purity solar grade material with low sinter ability. Silzot[®] HQ is a high-quality ceramic grade material with not optimized Al content. The developed powder qualities MV1, MV3 and MV8 are optimized powders with MV1 showing the best suitability for high thermal conductivity. The production of such optimized powders was carried out in large quantities on AlzChem's dedicated production facilities.

Table 2: Powder characteristics (XRF/ XRD) measured at IKTS

Parameter	amount / wt-%				
	MV1	MV3	MV8	HQ	SQ
Si_3N_4	99.93	99.90	99.92	99.91	99.99
Fe	0.01	0.03	0.03	0.03	-
Al	-	0.04	0.04	0.05	-
β - Si_3N_4 content	7.3 ± 0.1	12.4 ± 0.1	16.9 ± 0.1	14.5 ± 0.1	48.0 ± 0.2

2.2 Sintering

In order to achieve high thermal conductivity in Si_3N_4 ceramics, impurities in the raw powders must be minimized while maintaining good sintering activity. Especially the aluminum and oxygen [5] contents have to be considered. This was part of the development of a special grade Si_3N_4 powder by AlzChem (MV1). **Figure 1** shows that this powder quality allows to produce materials with high thermal conductivity which is in the same range as for the high quality E10 powder (UBE Corporation). The materials were sintered in a gas pressure furnace at 1880 and 1900 °C to generate an adequate microstructure [6]. Not only the aluminum content in the raw powder is relevant, but also the introduced impurities during processing. Such impurities caused by wear in the mill (appr. 0.2 wt.% Al) are the reason why during upscaling slightly lower thermal conductivities were determined, ref samples marked with "T" in **Figure 1**.

However, with an adapted processing, these impurities can be minimized. Additionally, the effect of impurities can be at least partially compensated by adaptation of the sintering temperature.

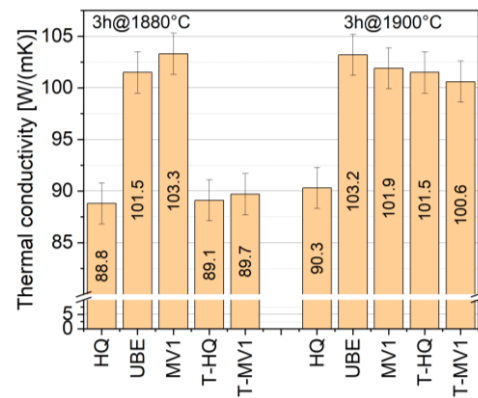


Figure 1: Thermal conductivity of the laboratory scale granules (80 g batches) made of HQ, UBE and MV1 raw powders and the small-scale production granules T-HQ and T-MV1 (10 kg batches) sintered at 1880 °C and 1900 °C, respectively.

The sintered large blocks (dimensions approx. $215 \times 145 \times 45$ mm), show a thermal conductivity of approx. $90 \text{ W}/(\text{m}\cdot\text{K})$, a biaxial strength of $> 850 \text{ MPa}$, a fracture toughness (SEVNB) of $6.9 \text{ MPa}\cdot\text{m}^{1/2}$ and a breakdown strength of $> 40 \text{ kV}/\text{mm}$. Sintered blocks of the ceramic were cut by multiwire sawing to $200 - 300 \mu\text{m}$ thick ceramic substrates at Fraunhofer CSP, **Figure 3**.

2.3 Manufacturing of ceramic substrates using multi-wire cutting

The properties of ceramic substrates for circuit boards are clear: high density – no holes or lunkers, good uniformity – no stoichiometric variations, and smooth surface with low total thickness variations (TTV) – no scratches, grooves or surface roughness. The requirement for lateral dimensions is in the range of 215×145 mm, substrate thickness should be $\leq 200 \mu\text{m}$.

In semiconductor industries, multi-wire cutting (see exemplary image in **Figure 2**) is the standard tool for wafer manufacturing.

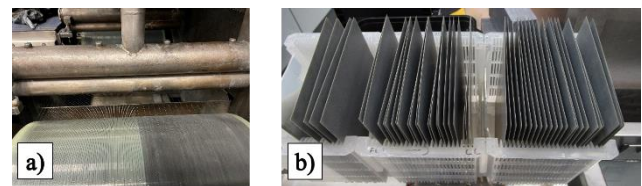


Figure 2: a) multi-wire arrangement (exemplary image, in the real process, the distance of the wires is significantly smaller), b) Batch of Si_3N_4 wafers ready for the cleaning step.

Further, the technique was greatly improved by photovoltaic production, today, billions of silicon wafers are produced by multi-wire cutting each year. For photovoltaic applications, the wafer size is now 210×210 mm, with a thickness of $150 - 200 \mu\text{m}$. Virtually, wafer thicknesses down to $> 100 \mu\text{m}$ are possible. In the case of silicon, one cutting process results in several thousand wafers. Compared to silicon, the challenge of cutting Si_3N_4 is the

significantly higher hardness: the ceramic is some 2 to 3 orders of magnitude harder than silicon. This requires low cutting speeds, resulting in process times of some 30 to 50 hours per cut. Thus, extremely stable process conditions are mandatory to achieve Si_3N_4 -wafers with low variations in their thickness.

At Fraunhofer CSP, Meyer-Burger multiwire-wire saws are used (models DS-264 and DS-265), using slurry-based processes with loose particles (SiC , B_4C , diamonds), as well as diamond-wire based cutting. Within the project, the cutting of full size Si_3N_4 master cards of 215×145 mm was achieved successfully, ref. **Figure 3**. With the current technique, some 200 substrates are realized within one cut. Both, the maximum number of wafers produced at a time as well as the minimum thickness achievable will be the tasks for further research.



Figure 3: green and sintered Si_3N_4 blocks and multi wire cut substrate of 7.5×5.5 inch and $300 \mu\text{m}$ thickness.

2.4 Copper qualities for AMB substrates

For AMB substrates, the quality of copper is a key factor. Several core requirements have to be met:

- high electrical and thermal conductivity for basic functionality of the end product,
- a high thermal stability of the copper's microstructure to keep desirable properties after active metal brazing with its substantial thermal load, up to $950 \text{ }^\circ\text{C}$,
- a high bond strength between the copper and ceramic sheets.

The most promising copper materials lie within the range of high purity copper (Cu-OFE: copper, oxygen free electronic grade) with highest conductivity to low-alloyed high-performance coppers with very good conducting properties. Alloying can increase thermal stability by impeding grain boundary mobility, which is required for recrystallization and grain growth. For a high bond strength of the substrate, a very ductile copper material might be most suitable as it can accommodate the much lower thermal expansion coefficient of Si_3N_4 ceramic.

Based on these requirements, several copper materials were investigated. Initially, laboratory scale batches were cast and processed, and the materials were characterized as produced and after an annealing step simulating the AMB

process. Subsequently, six different copper qualities were produced on an industrial scale and characterized intensively chemically, mechanically, and metallographically. Especially grain growth during the heat treatment of joining and joining strength to the ceramic substrates were of major importance.

It was found that a Cu-OFE variant with a homogeneous microstructure of a medium grain size ($300 - 600 \mu\text{m}$) best met the requirements. Its superior bond strength was demonstrated through peeling tests of model AMB substrates with $300 \mu\text{m}$ thick Cu-sheets on $320 \mu\text{m}$ Si_3N_4 . In peeling tests, a strip of copper is peeled off in a 90° angle from the ceramic. The tests are suited to get information about the strength of the metal to ceramic joints. The unit is N per mm width of the peeled copper strips.

At Peeling tests Copper with homogeneous medium grain size showed better results than standard Cu-OFE with an inhomogeneous coarse microstructure and the investigated high-performance alloys with a fine to ultra-fine microstructure, ref. **Figure 4**.

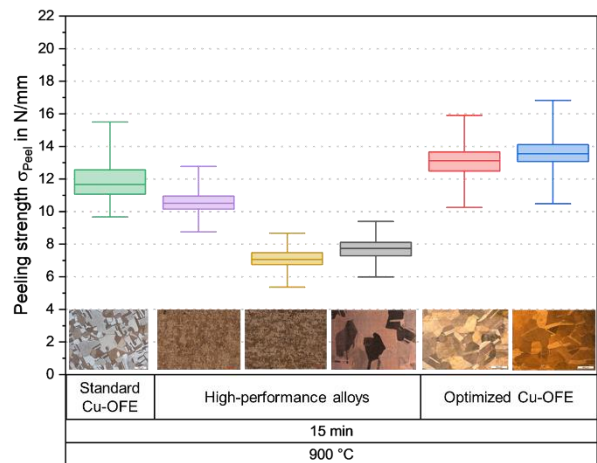


Figure 4: Peeling strength of different qualities of $300 \mu\text{m}$ Cu sheets joined by AMB on Si_3N_4 substrates. The corresponding microstructure is shown as well.

2.5 Active filler metal pastes

For joining the copper to Si_3N_4 , commercial active filler metal pastes have been tested [7-9] and new brazing pastes have been developed to achieve a better processability by screen printing, thinner layers and a homogenous distribution of minimized contents of the active phase TiH_2 . Suitable joining alloys are found in the Ag-Cu system. For fine powders of hypereutectic compositions, there are only few suppliers in Europe. A main drawback is the coarse particle size distribution of the used titanium source. To get homogenous and thin joining regions of $< 30 \mu\text{m}$, active filler metal pastes need to contain sufficient fine powder qualities.

In standard commercial AMB pastes coarse TiH_2 powder is used, see **Figure 5 a**). For a better distribution of the active component a finer powder was found, ref. **Figure 5 b**).

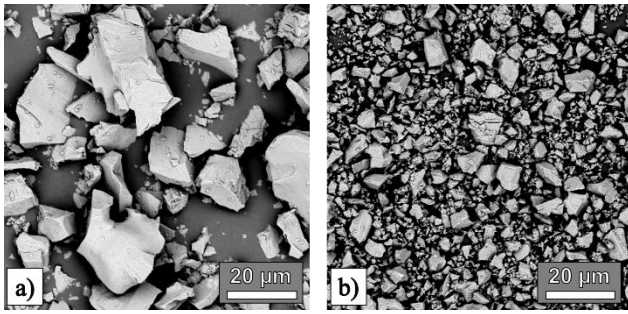
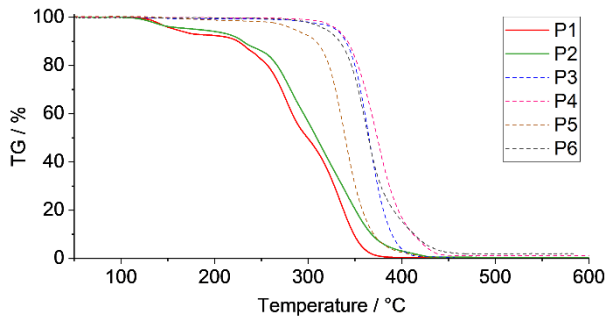


Figure 5: a) coarse standard powder of TiH_2 and b) fine quality of TiH_2 .

Also, the metal powder itself as the main component was substituted by a finer AgCu powder so at the end the brazing layer could be thinned from approx. 100 μm to less than 40 μm . With this step even material costs could be reduced. For the formulation of brazing pastes for joining areas with low porosity, the organic binder composition is of high importance. Together with the anorganic AgCu and TiH_2 the brazing pastes, processable by screen printing without any clogging of the mesh, have been formulated. Besides the Ag containing filler metals, also Ag-free compositions were made, based on Cu-Si-Ti-Al alloys. Thermal decomposition of different polymers was analyzed under nitrogen atmosphere. In

Figure 6 the data of the differential thermal analysis in combination the data from thermogravimetry shows the behavior of 6 different polymers. The starting point of the decomposition varies from 140 – 350 $^\circ\text{C}$ and shows single step, fast decomposition (polymers 3 – 6) above 310 $^\circ\text{C}$ as well as a slower decomposition (polymers 1 – 2) beginning



at 140 $^\circ\text{C}$.

Figure 6: Decomposition behavior of 6 different polymers in inert atmosphere.

Polymers 1 and 2 were chosen for the development of the brazing pastes. Based on these both polymers and a compatible solvent Ag-free and Ag-containing brazing pastes were manufactured.

For the AMB process, the presence of the active component Ti at the interface of the ceramic is essential to create a wettable layer on the ceramic surface during the soldering process. A fine distribution depends on the particle size of the TiH_2 as also on the utilized amount.

For Ag-containing pastes the TiH_2 content was varied in the range of 2.5 – 5.0 wt%. As can be seen from the cross

sections in **Figure 7** samples made of pastes containing 4.0 and 5.0 wt% TiH_2 show less pores in the brazing layer, then the other two samples.

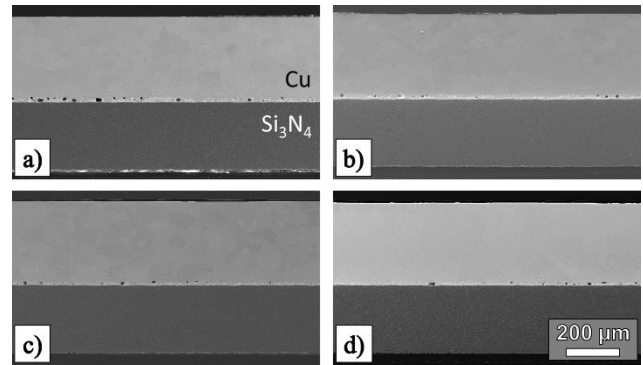


Figure 7: Cross sections of samples joined with brazes of different TiH_2 contents, a) 2.5 wt% TiH_2 , b) 3.0 wt% TiH_2 , c) 4.0 wt% TiH_2 , d) 5.0 wt% TiH_2 .

Also, the formation of Ti_xSi_y -phases, which leads to the embrittlement of the joining zone, was minimized by using the fine TiH_2 -powder in the paste formulations. Finally, the 5.0 wt TiH_2 containing filler metals was used for all further experiments for the AgCu AMB.

Active silver-based filler metals in AMB substrates are known to have a tendency to form silver dendrites when the corresponding modules are subjected to very high power densities. Within CuSiN the potential of activated Copper based filler metals to join Copper and Si_3N_4 has been evaluated. A similar study as for the silver based filler metals was done for Ag free Copper based brazing pastes, with best results in terms of bonding strength and pore free bonding layer for 3.0 wt % TiH_2 plus fine TiNi alloy powder and a joining temperature of 1050 $^\circ\text{C}$ (see **Figure 8 a**)

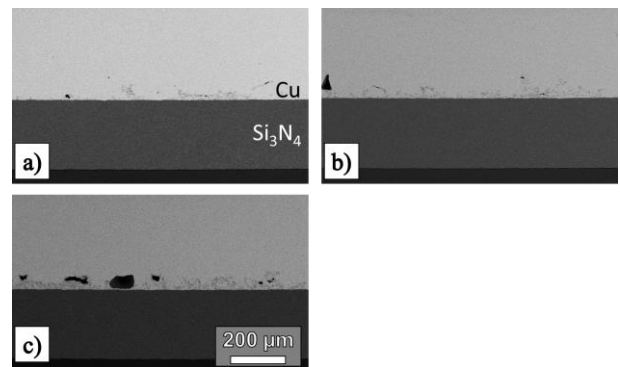


Figure 8: Ag free AMB joining with different TiH_2 contents, a) 3.0 wt% TiH_2 , b) 5.0 wt% TiH_2 , c) 8.0 wt% TiH_2 .

To gain active metal brazed Cu- Si_3N_4 -substrates with high bonding strength, the time-temperature regimes for organic decomposition and joining have to be developed and the quality of the joined components have been analyzed. The characterization was also performed by peeling tests, ref **Figure 9**. Highest peeling strength of about 13.5 N/mm are gained with Ag-containing active filler metals, lower brazing temperatures of 900 $^\circ\text{C}$ and soaking times < 10 minutes. A different dependency was found for Ag-free brazing pastes. Here a temperature of 1050 $^\circ\text{C}$ is

required to achieve a maximum peeling strength of 11 N/mm.

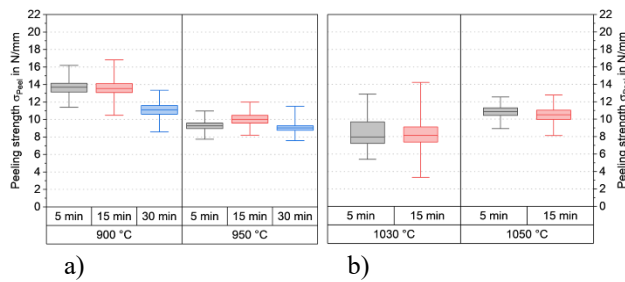


Figure 9: peeling strength of a) silver containing b) silver free active filler metals for joining 300 μm Cu-sheets to Si_3N_4 .

To investigate the difference in peeling strength, cross sections have been prepared and analyzed. Especially the formation of the bonding interface, made of TiN and brittle inclusions made of Ti_xSi_y have been analyzed as also the impact of silver in the joining region, ref. **Figure 10** and **Figure 11**. To the interface of the Si_3N_4 , a dense and thin bonding layer of TiN has been formed. At the chosen joining conditions. The thickness of this layer is below $0.5 \mu\text{m}$. On top of this layer crystals of Ti and Si are found, being titanium silicides of several composition and so marked as Ti_xSi_y . Due to the small crystal size of less than $2 \mu\text{m}$, an accurate determination of the Ti : Si ratio was not possible and so remains to be done in further investigations. The Ti_xSi_y -crystals also are detectable in the Ag-Cu alloy of the filler metal. Titanium silicide as brittle material shall be minimized at the interface being responsible for propagating of cracks during (thermo) mechanical load. In this context single Ti_xSi_y -crystals embedded in a more ductile metallic matrix are advantageous, because by deformation of the matrix, stress at the interface is reduced and cracking can be prevented.

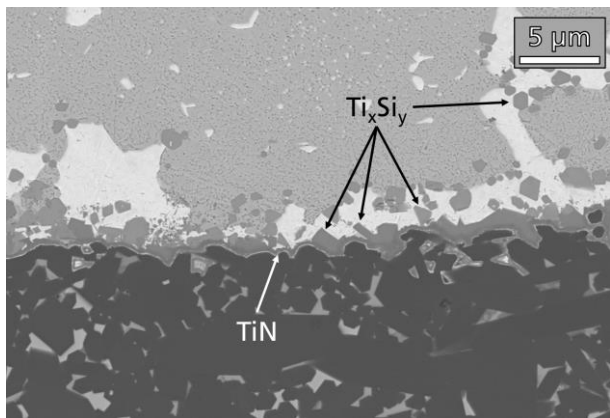


Figure 10: Interface of silver-based active filler metal and Si_3N_4 ceramic (bottom region in the figure)

Due to the much higher Cu-Content of the silver-free filler metals, the microstructure at the interface to Si_3N_4 ceramics differs significantly. In contact with the ceramic, again a $0.5 \mu\text{m}$ bonding layer of TiN has been formed. On top of this, several but single Ti_xSi_y crystallites are detectable. The filler metal alloy is quite homogeneous, not divided in

several components. In the copper alloy few Ti-rich crystals are embedded.

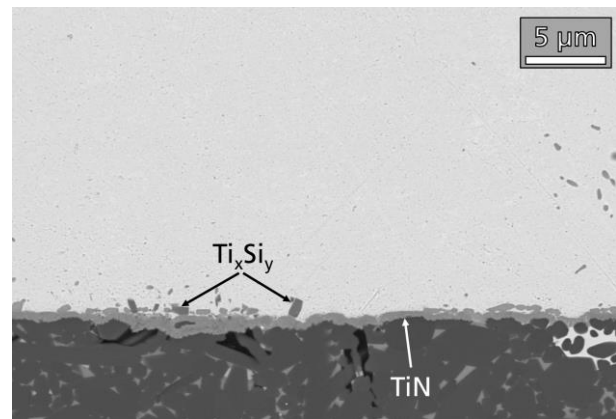


Figure 11: Interface of silver-free active filler metal and Si_3N_4 ceramic.

In comparison of the interfaces of both filler metal types, it is obvious that the amount of Ti_xSi_y crystals for Ag-based filler metals (**Figure 10**) is higher as for the Ag-free (**Figure 11**). In this context it is surprising, that the peeling strength of Ag-based filler metals is higher, ref Figure 9. We assume, that for the chosen joining conditions consisting of filler metal, temperature and joining components not the amount of Ti_xSi_y at the interface determines the strength, but the more ductile Ag-Cu matrix of the Ag-containing filler metal composition. So, by crack propagating, the Ag-Cu matrix allows a higher degree of deformation compared to the Ag-free one and so compensates the brittle properties of the Ti_xSi_y crystals at the interface. As result of the comparison of the interface layers of both filler metal types, it can be stated that more ductile phases at the joining interface leads to higher strength as only the reduction of brittle phases. So, at the chosen joining conditions the mechanical properties of Ag-based filler metals exceed those of silver free.

2.6 Concept for an industrial furnace system for vacuum brazing

As a part of the CuSiN project, a vacuum brazing furnace and a batch carrier system were conceptualized with the following objectives:

- Cost-efficient
- High vacuum pumping station
- Leak rate of the furnace: $< 1 \cdot 10^{-3}$ mbar \cdot l/sec
- Maximum vacuum in empty, clean, cold furnace: $< 5 \cdot 10^{-6}$ mbar
- High temperature uniformity $\pm 5^\circ\text{K}$
- Heating rate 10 K/min
- Maximum process temperature 950°C
- Almost carbon-free atmosphere
- Process time for brazing cycle less than 24 hours
- Batch size 400 to 500 mastercards
- Handling of high amounts of organic binder induced by the filler metal paste
- Batch carrier system with the lowest possible thermal dead load

- Hysteresis-free application of joining force to the mastercards

Based on the requirements of the involved project partners and their demands on the furnace and the process, a vacuum brazing system with a vertical design was developed over several engineering steps, concepts and simulations. The vertical design offers the major advantage that the system can also be under inert gas during charging and is only minimally contaminated by air and ambient humidity, thus contributing to optimize the process time for a brazing cycle.

In order to minimize the influence of the graphite heating zone, consisting of insulation and heater, a retort made of a nickel-based alloy was engineered and constructed. The functionality of this retort was demonstrated in initial tests in furnace chambers.

For further minimization of thermal loads, a CFC and graphite batch carrier system was developed, implemented and tested ref **Figure 12**. With this carrier system, the load required for aligned brazing of the AMB-substrates is not applied by dead loads, but by a spring system. The spring system is space saving and can be customized to the desired parameters. It has been successfully tested in several trials in industrially scaled furnaces operated by PVA LWT.

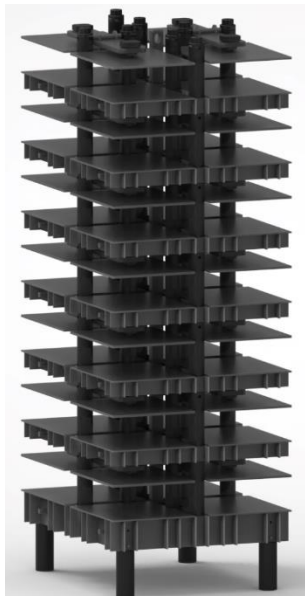


Figure 12: Modular batch carrier for up to 560 Mastercards.

The vacuum furnace system as also the batch carrier fulfills all necessary requirements. A customized production could be started on requirement.

2.7 Process transfer to master card size

Based on the joining and process development results in lab scale furnaces, an upscaling to large scale substrates of master card size was carried out by PVA LWT. A multi-

purpose vacuum furnace ($1.900 \times 600 \times 1.000$ mm; $L \times W \times H$), equipped with a multistage high-vacuum pumping station was used for these experiments:

For the up-scaling approach following main aspects had to be considered:

2.7.1 Debinding:

Due to the larger joining area, high quality requirements as well as complex structures, special emphasis had to be set onto removal of the organics in the screen-printed paste. Based on the generic data of the binder, the debinding temperature was set to $450\text{ }^{\circ}\text{C}$, with a soak time of 120 min. Two atmospheric regimes were analyzed – debinding under high vacuum and at 1 mbar partial pressure of 5.0 Argon.

Under vacuum conditions a distinctive rise of the system-pressure was observed. Due to the large recipient volume, combined with a corresponding pumping power, the rise was limited to approx. 25 minutes and the pressure level did not rise above $1 \cdot 10^{-3}$ mbar. At the end of the soaking time a pressure level below $1 \cdot 10^{-4}$ mbar was achieved. The same final vacuum level was achieved using inert gas purging.

2.7.2 Chemical interaction between carrier and substrate:

Due to the titanium content in the brazing filler a suitable carrier material had to be used. Even though graphite or CFC are very commonly used for brazing tooling purposes, the interaction between the carbon and titanium had to be investigated. First brazing samples in the CFC carrier has been used for SEM-EDX Analyses. As can be seen in the **Figure 13**, the interaction between Carbon and Titanium is limited only to the surface area of the brazing gap (meniscus) and no penetration between the joining partners could be identified. Additionally, no significant Titanium depletion took place and the active brazing filler metal still wetted the sample ceramics.

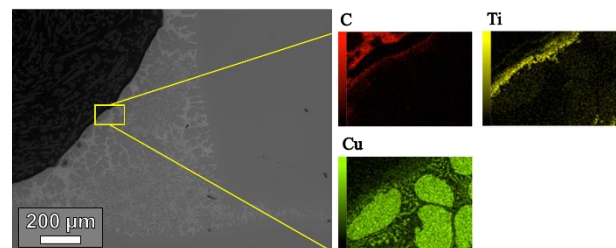


Figure 13: Cross section of filler metal surface and EDX scan of the area

2.7.3 Mechanical interaction between carrier and substrate:

For planar joining interfaces with thin materials such as in the case of the mastercard substrates a homogeneous force has to be applied. Not sufficient force and force gradients can lead to distortion, delamination and voids, consequently resulting in a reduced yield of production.

Two different designs of substrates have been used to show the capabilities of the brazing filler paste, the derived parameters and the carrier – a strip design for mechanical testing purposes and a geometrical design with application relevant features such as narrow pitches between the connected structures.

In order to visualize the results, scanning ultrasonic microscopy was used. Using just the pressing force of the clamping device of the carrier with a thin distribution plate led to an uneven loading of the planar interface. As can be seen in the **Figure 14** the central area of the substrate was brazed without significant voids. Nevertheless, larger delamination areas can be observed on the outer edges and especially the corners.

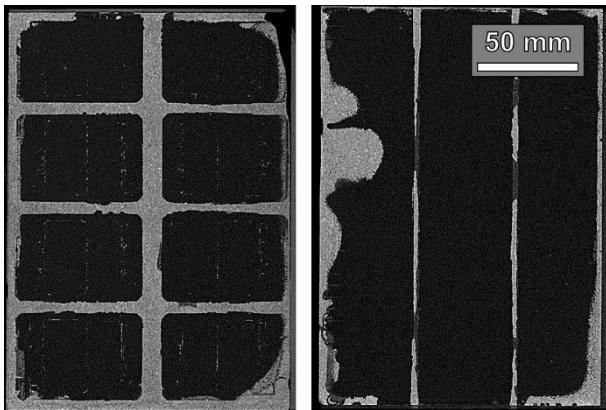


Figure 14: scanning ultrasonic microscopy image of test joinings in mastercard size.

Additionally, it can be stated, that the brazing melt spread onto areas where geometrical features should remain free (pitches). Especially, narrow geometrical features have been influenced by this effect.

In **Figure 15** an ultrasonic image of substrates after improvements of the set-up and the parameters (more rigid set-up, reduction of the clamping force and the brazing temperature and soak time) is depicted. The delamination magnitude on the edges and the corners was reduced significantly. The optimization of the processing parameters led to more pronounced net-shape behavior of the flow kinetics of the brazing filler metal.

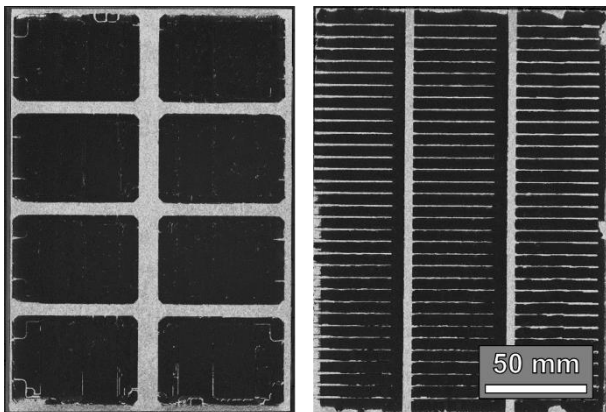


Figure 15: scanning ultrasonic microscopy image of mastercards after optimization of the joining process.

Even though further improvements are necessary for an industrial scale brazing, the promising results of the proof-of-concept work show the possibilities of the brazing set-up, the filler metal and the whole material chain.

3 Summary

In a German consortium the whole value chain for producing substrates for Cu-Si₃N₄ circuit boards was implemented. High quality Si₃N₄ raw powder were processed to dense ceramic blocks and ceramic wafers were cut by multiwire sawing. The ceramic wafers show high thermal conductivity of up to 90 W/(m·K) and superior biaxial strength of > 850 MPa.

With suited copper qualities Cu-Si₃N₄ components were joined with silver containing and also silver free AMB processes. This process successfully was transferred from laboratory scale to an industry scale of mastercard size. Additionally, an industry suited vacuum furnace system for production was developed and a batch carrier for mastercards developed and successfully tested.

4 Acknowledgement

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